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METHODS FOR DETERMINATION OF ALDICARB RESIDUES IN CROPS, SOIL AND WATER

UDC 664 : 543 [632·95·028 ALDICARB]

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Indian Standard

METHODS FOR DETERMINATION OF ALDICARB RESIDUES IN CROPS, SOIL AND WATER

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(*Continued on page* 2)

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AMENDMENT NO. 1 MARCH 1985

TO

IS:10629-1983 METHODS FOR DETERMINATION OF ALDICARB RESIDUES IN CROPS, SOIL AND WATER

(*Page 5, clause 3.4, heading*) – Substitute 'Clean-Up' *for* 'Cleen-Up'.

(*Pages 5 and 6, clause 3.4, line 20*) – Substitute '10 ml' *for* '100 ml'.

(*Pages 5 and 6, clause 3.4, line 25*) – Substitute 'sodium hydrogen carbonate' *for* 'sodium carbonate'.

(AFDC 56)

Reprography Unit, ISI, New Delhi, India

Indian Standard

METHODS FOR DETERMINATION OF ALDICARB RESIDUES IN CROPS, SOIL AND WATER

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 August 1983, after the draft finalized by the Pesticides Residue Analysis Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 Aldicarb has a broad spectrum of effectiveness against many species of insects, mites and nematodes. It is systemic and applied only in the form of granules and is used as a soil treatment on a range of crops including bananas, coffee, tobacco, cotton, potatoes, sweet potatoes and sugarcane. Frequent and increased use of aldicarb formulations often result in harmful effects due to toxic nature of residues. Careful assessment of residues is, therefore, an important step in safeguarding human health and in the establishment of sound regulatory policy.

0.3 This standard will enable the health authorities and others to follow uniform test procedure for the estimation of aldicarb residues in various crops, soil and water.

0.4 In reporting the result of a test or analysis made in accordance with this standard if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes spectrophotometric and gas chromatographic methods for the determination of aldicarb [2-Methyl-2-(methylthio) propionaldehyde 0-(methylcarbamoyl) oxime] residues in crops, soil and water. The residues determined include aldicarb, its sulfoxide and the sulfones.

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*Rules for rounding off numerical values (*revised*).

1.1.1 Spectrophotometric method may be adopted as a limit test for routine purposes with the minimum detection limit of 0·1 /µg/g (0·1 ppm) whereas gas chromatographic method shall be the reference method with the minimum detection limit of 0·005/µg/g (0·005 ppm).

2. SAMPLING

2.1 The representative samples for the purpose of estimating aldicarb residues in various crops, soil and water shall be in accordance with the sampling procedures as prescribed in the relevant Indian Standards, wherever available.

2.2 Preparation of Laboratory Sample

2.2.1 *Soil, Coffee, Tobacco, etc* — If needed, grind to pass through suitable sieve. Reduce to about 100 g by mixing and quartering.

2.2.2 *Vegetables and Fruits*

2.2.2.1 Remove visible soil particles by gentle rubbing. Root vegetables may need scrubbing with a brush and some washing.

2.2.2.2 Remove all inedible portions of vegetables and stems and stones of fruits if any.

2.2.2.3 If a peel or skin is not normally consumed, it should be removed and if edible it should be included.

2.2.2.4 Cut a representative sample into pieces and reduce by mixing and quartering to about 300 g.

2.2.2.5 Blend the 300 g sample in a waring blender to obtain homogeneous mixture. Mix content with sepatula and reblend to ensure homogeniety.

2.2.3 *Water* — This does not need any processing.

3. EXTRACTION AND CLEAN-UP

3.1 Apparatus

3.1.1 *Rotary Flash Evaporator*

3.1.2 *Waring Blender or Equivalent* — Leakproof and explosion proof.

3.1.3 *Chromatographic Column* — 30 cm length and 1·3 cm inner diameter.

3.1.4 *Water Bath*

3.1.5 *Air Condenser* — Snyder type

3.1.6 *Magnetic Stirrer cum Hot Plate*

3.2 Reagents

3.2.1 *Acetone* — Analytical reagent grade/glass redistilled.

3.2.2 *Acetonitrile* — Analytical reagent grade/glass redistilled.

3.2.3 *Chloroform* — Analytical reagent grade/glass redistilled.

3.2.4 *Hyflo Super-Cel*

3.2.5 *Methanol* — Analytical reagent grade/glass redistilled.

3.2.6 *Benzene* — Analytical reagent grade/glass redistilled.

3.2.7 *Sodium Sulfate* — Anhydrous, analytical reagent grade.

3.2.8 *Glacial Acetic Acid* — analytical reagent grade.

3.2.9 *Hydrogen Peroxide* — 30 Percent.

3.2.10 *Coagulating Solution* — Prepared by dissolving 0·5 g of ammonium chloride in 400 ml of water containing 1 ml of orthophosphoric acid.

3.2.11 *Florisil* — 150 -250 µm (60 -100 mesh).

3.3 Extraction — Transfer 50 g of sample to a blender. Add 150 ml of an 80 : 20 mixture of acetone-chloroform and 100 g of anhydrous powdered sodium sulfate. Blend for two minutes at low speed and allow to settle for one minute. Decant the solvent into a Buchner funnel containing Whatman No. 1 filter paper covered with a thin coat of hyflo-supercel attached to a 500 ml filter flask. Apply vacuum cautiously until all the solvent has filtered into the filter flask. Re-extract the cake alongwith the filter paper with two 100 ml portions of acetone-chloroform and filter the extracts. Wash the blender and cake with 50 ml of mixed solvent.

3.4 Cleen-Up — Transfer the filtrate (*see* 3.3) to a 500 ml round bottom flask and add one drop of diethylene glycol. Connect the flask to a rotary vacuum flash evaporator and place in a water bath at 30°-40°C. Apply vacuum and reduce the pressure carefully to about 50 mm Hg. After the solvent has evaporated, disconnect the flask immediately. Add 5 ml of acetone, swirl and warm the flask under hot tap water for 30 seconds. Add 50 ml of coagulating solution and swirl the flask. Allow mixture to stand for 30 minutes with occasional swirling. Filter using vacuum, through 3 mm layer of hyflo-supercel on Whatman No. 1 filter paper in a Buchner funnel into a receiving flask. Wash the flask and precipitate with 2×25 ml portions of methanol-water (1 : 9), allowing each washing to remain in contact with precipitate about 20 seconds before applying vacuum. Transfer the filtrate to a 250 ml separatory funnel and add 30 ml of chloroform, shake well and allow the layers to separate completely. Drain the lower layer through a bed of anhydrous sodium sulfate into a 250-ml round bottom flask. Repeat extraction of aqueous layer with 2 × 30 ml portions of chloroform and collect the extracts in the same 250-ml flask. Wash the sodium sulfate bed with 2 × 20 ml portions of chloroform. Add one drop of diethylene glycol and evaporate to dryness under vacuum as before. Add 100 ml of hydrogen peroxide-glacial acetic acid (2 : 1) to the residue flask, attach an air condenser (snyder type) and oxidize the residues with continuous stirring at 70 ± 2°C for 30 minutes on a magnetic stirrer-cum-hot plate. Remove the flask and cool in an ice bath for 5 minutes. Add 60 ml of 10 percent aqueous sodium carbonate to neutralize the mixture. Transfer the neutralized mixture to a 250-ml separatory funnel and extract with chloroform as before. Add one drop of diethylene glycol and evaporate to dryness under vacuum as before. The residue is dissolved in 100 ml of 10 percent acetone in benzene poured on to a 12·5 cm column of florisil contained in a 13-mm i.d. glass chromatograph tube, the florisil being prewet with benzene. The column is allowed to elute and the eluate is discarded. This discard is necessary to remove the nitrile and oxime metabolities of aldicarb, which would interfere in the aldicarb determination. Finally, the aldicarb residues now oxidized to aldicarb sulfone, are eluted from the column in 150 ml of acetone-benzene (40 : 60).

4. SPECTROPHOTOMETRIC METHOD

4.1 Principle — The determination of total aldicarb residue is based on the carbamoyl-oxime group of the molecule. The carbamoyl oxime is hydrolyzed with base to 2-methyl-2-(methylthio) propionaldehyde oxime. Hydrolysis of the oxime in acidic medium forms the aldehyde 2-methyl-2-methylthio propionaldehyde and hydroxylamine. The hydroxylamine is oxidized with iodine to nitrous acid, which is determined colorimetrically at 530 nm.

4.2 Apparatus

4.2.1 Spectrophotometer — Capable of measuring at 530 nm.

4.3 Reagents

4.3.1 *Sodium Hydroxide* — 0·1 N aqueous solution.

4.3.2 *Hydrochloric Acid* — 1 N solution.

4.3.3 *Sulfanilic Acid* — Dissolve 1·0 g in 75 ml of water and add 25 ml of acetic acid.

4.3.4 *Iodine* — 1 percent in acetic acid (*m/v*).

4.3.5 *Sodium Thiosulfate* — 2 percent in water.

4.3.6 *Potassium Acetate* — Dissolve 100 g in 100 ml of water.

4.3.7 *1-Naphthylamine* — Dissolve 0·5 g in 50 ml of acetic acid, add 115 ml of water and 25 mg of activated charcoal, mix well and filter. Prepare fresh daily just befor use.

4.4 Estimation of Aldicarb Residues — Place an appropriate aliquot (*see* **3.4**) in a 250-ml flask, add one drop of diethylene glycol and evaporate to dryness as before. Add 5·0 ml of 0·1 N sodium hydroxide and place the flask in a water bath at 40°C for exactly 40 minutes. Add 1·0 ml of 1N hydrochloric acid. Place the flask in a boiling water bath (80-85°C) and allow to digest for 20 minutes. Allow the solution to cool in an ice bath for 10 minutes and add 0·5 ml of sulfanilic acid solution. Mix well and add 0·2 ml of iodine solution, mix and let stand for 3 minutes. Add 0·2 ml of potassium acetate solution, mix and add 0·2 ml of sodium thiosulfate solution. Immediately add 0·3 ml of 1-naphthylamine solution and mix well. Let stand 1 minute and dilute to 10 ml with (1 + 1) acetic acid-water solution. Mix and let stand 5 minutes. Transfer the solution to a 125-ml separatory funnel and extract with 5 ml of chloroform. Let the layer separate completely and determine the absorbance of the top layer (aqueous) in a 1-cm cell at 530 nm, using a reagent blank processed along with the sample. Determine the concentration of aldicarb sulfone from a curve obtained by plotting μg of the standard in ml against absorbance.

4.4.1 *Preparation of Standard Curve* — Weigh 50 mg of aldicarb sulfone, transfer to a 100-ml volumetric flask and dilute to volume with acetone. Take an aliquot of 10 ml and transfer to 100-ml volumetric flask and dilute to volume with acetone. Dilute the second solution the same way and this gives a concentration of 5·0 μg per ml. Pipette aliquot of 0, 1.0, 2·0, 4·0, 6·0, 8·0 and 10·0 ml into a series of 20-ml test tubes and make the volume to 10 ml with chloroform. Follow the same procedure as described under **3.4** and **4.4**. Plot the absorbance of the standard solutions against μg of aldicarb sulfone to obtain a standard curve.

4.5 Calculation

$$\text{Aldicarb residues } \mu\text{g/g (ppm)} = \frac{\mu\text{g aldicarb sulfone} \times \text{dilution factor}}{\text{mass of sample (g)}}$$

4.6 Expression of Result — Report total toxic aldicarb residues as aldicarb sulfone in the sample as μg/g (ppm).

5. GAS CHROMATOGRAPHIC METHOD

5.1 Principle — The toxic residue of aldicarb in biological substrates is composed of aldicarb and its sulfoxide and sulfone metabolites. All three of these components are determined as a total residue by first oxidizing aldicarb and aldicarb sulfoxide to aldicarb sulfone with hydrogen peroxide-glacial acetic acid (2 : 1) and then determining total aldicarb sulfone by gas chromatography. The aldicarb sulfone is determined utilizing a flame photometric detector incorporating a 394-filter specific for sulfur-containing compounds, and it is quantitated by reference of the peak area to a previously prepared calibration curve.

5.2 Apparatus

5.2.1 *Gas Chromatograph* — Equipped with flame photometer detector incorporating a 394 filter specific for sulphur. The gas chromatograph is operated under the following suggested parameters. These parameters may be varied as per available facilities provided standardization is done.

Column	A stainless steel, glass column of 2·4 m length and 3·13 mm ID packed with chromosorb P coated with 5 percent carbowax 20 M.
Column oven temperature	180°C
Injection port temperature	300°C
Detecter temperature	200°C
Carrier gas	Nitrogen 60 Hydrogen 30 Air 30 ml per minute
Recorder chart speed	1 cm/minute

5.3 Procedure

5.3.1 Transfer suitable aliquot (*see* **3.4**) to a 100 ml flask and evaporate to dryness. Dissolve the concentrated residues in 1 ml of acetone and inject 8 μl into the column using a microlitre syringe. Identify the aldicarb sulfone peak by its retention time and measure the peak area.

5.4 Calculation

$$\text{Aldicarb sulfone } \mu g/g \text{ (ppm)} = \frac{As}{Astd} \times \frac{M}{M_1} \times \frac{V}{V_1} \times f$$

where

As = area sample;

$Astd$ = area standard;

M = μg of standard injected;

M_1 = mass, in g, of the sample;

V = volume of final extract in ml;

V_1 = μl of the sample injected; and

f = recovery factor = $\dfrac{100}{\text{percent mean recovery}}$

INDIAN STANDARDS ON PESTICIDES RESIDUE ANALYSIS

IS :

5863 (Part I) - 1970	Method for determination of malathion residues: Part I Cereals and Oilseeds
5864-1983	Method for determination of DDT residues in food commodities (*first revision*)
5952-1970	Method for determination of parathion residues in foodgrains and vegetables
6169-1983	Methods for determination of BHC (HCH) residues in food commodities (*first revision*)
10168-1982	Methods for determination of fenitrothion residues in foods
10169-1982	Methods for determination of carbaryl residues in fruits and vegetables

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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